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Mitigation of autogenous shrinkage in repair mortars via internal curing

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Abstract: Repair mortars are being used with increasing frequency to maintain the aging US infrastructure. Durability is a key concern for such repair materials and both their volume stability and bond to the existing concrete are key attributes, the former to avoid excessive cracking of the repair and the latter to ensure that it remains in place as an integral part of the (repaired) concrete structure. This paper examines the volume stability of two commercially available repair mortars by measuring their autogenous deformation to an age of 28 d. Internal curing is examined as a strategy to mitigate the significant autogenous shrinkage encountered for both materials. The performance of pre-wetted lightweight aggregates (LWA), a superabsorbent polymer (SAP), and a superabsorbent polymer-coated sand (PCS) with respect to compressive strength and autogenous deformation are evaluated. Because these three internal curing agents have water absorptions spanning two orders of magnitude, they have differing influences on the proportioning of the mortars and on subsequent performance. In general, when proportioned with the same amount of internal curing water, the mortars based on LWA exhibit the highest compressive strengths and the greatest relative reductions in autogenous shrinkage in comparison to the controls formulated without internal curing.

1.0 INTRODUCTION

In the United States, much attention continues to be focused on the state of the nation's physical infrastructure, as exemplified by the report card that the American Society of Civil Engineers issues every few years. In 2013, the most recent report card provided an overall grade of a D+, with an estimated investment of $3.6 trillion needed by 2020.[1] It is clear that repair materials will play a critical role in the restoration and subsequent maintenance of the concrete structures (roads, bridges, pipes, etc.) comprising a significant part of this infrastructure. When a repair material is applied to an existing concrete substrate, not only must one consider the performance properties of the repair material, but also its compatibility with the existing concrete (material and structure). Early-age volume changes in the repair material can produce interfacial stresses that can lead to premature cracking and debonding of the repair, failing to produce the desired service life.

While repair materials are typically characterized with respect to their drying shrinkage, and many are formulated with special expansive agents to minimize this quantity, their volume changes under sealed conditions (autogenous deformation) can be equally important to their overall field performance. To date, however, autogenous deformation is rarely quantified and seldom reported on the manufacturer's materials specification sheets. De la Varga and Graybeal[2] have used the ASTM C1698[3] standard test method for measuring the autogenous deformation of grout-type materials. In their study, they also considered the mitigation of autogenous shrinkage in a few of their materials by supplying internal curing[4,5] (IC) via the utilization of pre-wetted lightweight aggregates (LWA). In the present study, two commercially available repair mortars are evaluated with respect to mechanical properties and autogenous deformation, and for each material, three different IC agents are investigated as a possible means to reducing autogenous shrinkage without sacrificing strength or modulus.

2.0 MATERIALS AND EXPERIMENTAL PROCEDURES

Two commercially available repair mortars were utilized in the present study. The first, designated as material S, is a "one-component, pre-packaged, ready-to-use, cementitious, silica fume, fiber reinforced, high strength shrinkage-compensated mortar." The second, designated as material M, is a "one-component, shrinkage-compensated, fiber-reinforced product that contains an integral corrosion inhibitor." For the control mortars without internal curing, the

mixing water was set at the middle of the manufacturers' recommended limits; for material S, this resulted in a water-to-solids ratio (*w/s*) by mass of 0.135, while the *w/s* for material M was slightly less at 0.129. It should be kept in mind that in a typical repair mortar (bag) mix, only 30 % to 40 % of the powder is reactive with water, suggesting a water-to-cementitious materials ratio (*w/cm*) by mass on the order of 0.32 to 0.45.

All mortars were prepared in a three-speed planetary mixer with a typical batch size being based on 3600 g of dry repair mortar. Following the manufacturers' recommendations, the following mixing procedure was employed:

1) 80 % of the mixing water was added to the mixing bowl along with the dry repair material,
2) mixing was conducted for 30 s on low speed,
3) the additional 20 % of the mixing water was added during an additional 30 s of mixing on low speed,
4) mixing speed was changed to intermediate and an additional 30 s of mixing was conducted,
5) the mortar was allowed to rest for a period of 90 s, during the first 30 s of which the sides of the mixing bowl were scraped down, and
6) mixing was concluded with 60 s of mixing on intermediate speed.

Fresh mortars were evaluated for mix temperature (range of 20 °C to 23 °C for the eight different mortars investigated in the present study), density via mass measurements of a filled 400 mL volumetric brass cup, and flow using a flow table (ASTM C1437[6]). For a few of the mixtures, in situ X-ray diffraction (XRD) was conducted on a single specimen of the fresh mortar during the first 24 h of its hydration, to provide an indication of phase development at early ages via subsequent Rietveld analysis. To provide enough material for all of the required specimens, each mixture was prepared in two individual batches in the same morning, with the measured values for density and flow for each batch being provided in Table 1.

The focus of the present paper is on the influence of IC on autogenous shrinkage, compressive strength, and elastic modulus for these two repair mortars. Autogenous shrinkage was evaluated by casting three corrugated tube specimens for each mixture and measuring their deformation during the first 28 d of sealed curing according to ASTM C1698.[3] Compressive strength was measured at 1 d, 7 d, and 28 d on mortar cubes prepared and tested according to ASTM C109.[7] Elastic modulus was measured using sonic (frequency) techniques (ASTM C1259[8]) on 2.54 cm by 2.54 cm by 27.94 cm hardened mortar prisms that had been exposed to drying conditions (23 °C ± 1 °C, 50 % ± 2 % RH) for 25 d, following 3 d of immersion in water saturated with calcium hydroxide.

For mixture proportioning with IC, the amount of IC water needed was determined according to the procedures outlined in Bentz et al.[9], by experimentally measuring the 28 d chemical shrinkage of the two control repair mortars using the ASTM C1608 standard test method.[10] For both mortars, a 28 d value of 0.0225 g water/g repair mortar (dry) was measured. IC was provided by three different means to each of the two repair materials, but in each case the same quantity of IC water given above was proportioned into the mixture.

For the first IC agent, an expanded shale LWA was obtained from a manufacturer and sieved through a #8 sieve to remove the coarser particles. The remaining fines were characterized with respect to their dry density (1410 kg/m^3), saturated-surface-dry density (1688 kg/m^3), absorption (20 % by mass of dry LWA), and desorption (98 % when exposed to a slurry of KNO_3, with a controlled relative humidity of about 92 % at 23 °C), as per the ASTM C1761 standard specification.[11] The LWA was added to the mortar in a pre-wetted condition, after equilibrating for at least 72 h.

The second means of providing IC was via the utilization of a superabsorbent polymer-coated sand (PCS). Based on information from the manufacturer and preliminary experiments, it was determined that the absorption of the PCS in a cement-based system was on the order of 2 g water/g dry PCS. The PCS would have a higher absorption when placed in distilled water,

but the value is reduced in the highly ionic, high pH solution that is typically found in mortars and concretes. The PCS was added to the mortar in a dry condition and the additional IC water was included as a part of the mixing water.

The third means of supplying IC was via the use of a superabsorbent polymer (SAP). The SAP consists of particles with a median particle size of 60 µm (laser diffraction), that swell to a diameter of about 400 µm when placed in distilled water. However, once again, the swelling is significantly less within a cement-based matrix. In this case, X-ray microtomography measurements and information from the manufacturer were used to determine an absorption on the order of 20 g water/g SAP (dry) for this material, approximately 10 times that of the PCS and 100 times that of the LWA. The SAP was pre-mixed dry with the repair mortar and the additional IC water was included as part of the mixing water.

The various means of supplying IC explored in this study are illustrated schematically in Figure 1. It is of interest to note that because the repair mortar is a pre-packaged material, the IC agent is added "on top" of the ordinary mixture proportions, altering the volume fraction of repair mortar in the mixtures with IC vs. the control mortars prepared without IC. This is especially noticeable for the repair mortars prepared with LWA, where 18 % of the final volume is occupied by the rigid LWA particles. The changes to mixture volume fractions illustrated in Figure 1 would be expected to also influence mechanical properties and autogenous shrinkage of the produced mortars.

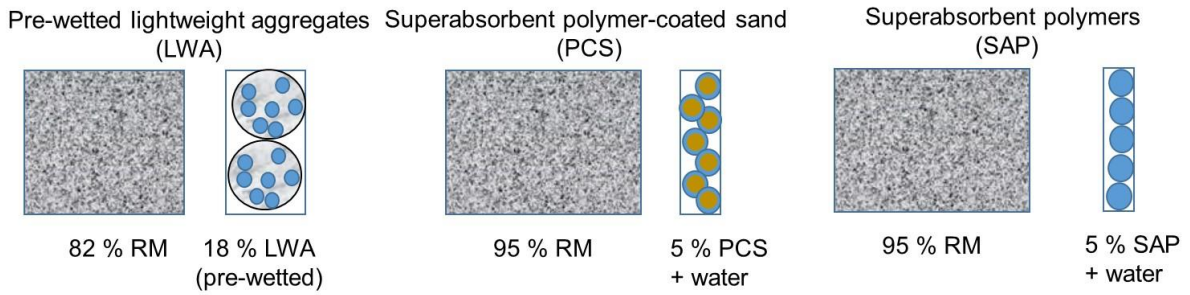

Figure 1. Illustration of repair mortar (RM) mixture proportioning (volume fractions) for internal curing with LWA, PCS, and SAP. Each mixture has been proportioned to provide an identical quantity of internal curing water per unit mass of the repair mortar.

3.0 RESULTS AND DISCUSSION

The properties of the fresh mortars and a summary of their measured mechanical properties is given in Table 1, along with baseline values taken from the manufacturers' specification sheets for the two repair mortars (without IC). In general, there is reasonable agreement between the fresh mortar densities and flow values measured on the two sub-batches for each mixture. Because of the additional water introduced into the mortars with IC, they exhibit a lower density than the corresponding repair mortar formulated without IC. In both cases, the performance of the control mortars prepared and evaluated under laboratory conditions exceeded the manufacturers' specified values. In general, the strength and modulus of the two materials are similar, with material S being slightly stronger/stiffer than material M.

Since IC curing introduces additional (initially water-filled) porosity into the mortar, in general, compressive strengths are reduced in comparison to the corresponding repair mortar without IC. This strength reduction is less severe in the case of the pre-wetted LWA, as the emptied LWA particles contribute to a rigid framework within the mortar and the increased porosity is somewhat offset by the enhanced hydration provided by IC.[4,5] The strength reductions are more severe in the cases of the PCS and SAP mortars, as the superabsorbent polymer particle (SAP) or shell around a sand grain (PCS) is replaced by (empty) porosity once its IC water is released to the surrounding hydrating cement paste. For these SAP-based materials, strength reductions are even more severe at 1 d in most cases, as these polymers also absorb (calcium) ions and produce a measurable retardation of the cement hydration reactions, as verified by isothermal calorimetry studies (not presented in this paper).

Table 1: Fresh and mechanical properties for repair mortars with and without internal curing. Cases where the measured values were below those provided on the manufacturers' specification sheets are highlighted in either yellow (≤ 20 %) or grey (> 20 %).

	S	S-LWA	S-PCS	S-SAP	M	M-LWA	M-PCS	M-SAP
Fresh density	2.12/2.15	2.09/2.09	2.10/2.11	2.06/2.10	2.11/2.21	2.02/2.00	2.04/2.02	2.03/2.03
Flow (table)	91/85	77/79	79/88	88/77	107/93	88/85	87/93	107/113
1-d strength (MPa)	38.8 (1.1 %)[A]	36.6 (2.0 %)	32.2 (5.6 %)	13.5 (4.6 %)	28.6 (1.3 %)	19.2 (6.3 %)	13.0 (10 %)	12.9 (7.1 %)
7-d strength	69.9 (5.9 %)	70.3 (1.3 %)	64.0 (1.0 %)	46.7 (4.6 %)	59.9 (19 %)	52.0 (3.7 %)	42.7 (7.4 %)	42.8 (2.2 %)
28-d strength	89.8 (6.6 %)	84.6 (5.2 %)	80.2 (6.2 %)	62.7 (8.3 %)	90.8 (5.6 %)	73.6 (2.5 %)	59.0 (3.3 %)	52.8 (4.0 %)
28+-d modulus (GPa)	36.8 (6.0 %)	33.7 (3.4 %)	30.3 (2.6 %)	28.8 (2.8 %)	34.4 (5.5 %)	31.6 (19 %)	29.3 (4.8 %)	27.7 (2.0 %)
Manufacturers' Specification Sheet Values								
1-d strength (MPa)	31				24.1			
7-d strength	55				45.5			
28-d strength	69				62.1			
28-d modulus (GPa)	Not listed				34.5			

[A]Standard deviation for three replicate specimens.

The autogenous deformation results measured over the course of 28 d for the eight mortar mixtures are provided in Figure 2. The presented measurements were initiated at a time zero corresponding to the final setting time of each mortar mixture (varying between 5 h and 8 h), as determined using needle penetration (results not presented in this paper). For the repair mortars without internal curing, material S exhibits a slight expansion for the first day, followed by monotonic shrinkage, while material M exhibited only shrinkage over the course of the 28 d measurement. The microstrain measured after 28 d, about 350 for material S and 520 for material M, are significant and would suggest that these materials might experience early-age cracking under field conditions. As expected, internal curing reduced the autogenous shrinkage, but to different degrees depending on the internal curing agent being employed. In Figure 2, one can observe that the initial expansion in the mortars with internal curing depends on the agent employed, with greater expansions obtained with the PCS and SAP than with the LWA. As shown schematically in Figure 1, this is partially due to the additional restraint provided by the 18 % LWA in the latter mortars. This is also consistent with the lower measured modulus (more deformable) for the PCS and SAP mortars as shown in Table 1. Based on the quantitative XRD measurements provided in Figures 3 and 4, much of this early-age expansion is likely due to ettringite formation, which tends to grow as needles that can "push open" the microstructure. This same expansive formation of ettringite is occurring in the mortars without internal curing, but in that case, it is mostly offset by the autogenous shrinkage due to self-desiccation accompanying the chemical shrinkage produced by the ongoing hydration reactions.[4] The measured autogenous deformation is always a balance of the self-desiccation-induced autogenous shrinkage with any expansion produced by the ongoing chemical reactions, typically due to ettringite formation. In the systems with internal curing, the self-desiccation is eliminated or vastly reduced, so that more of this "natural" expansion can be observed.

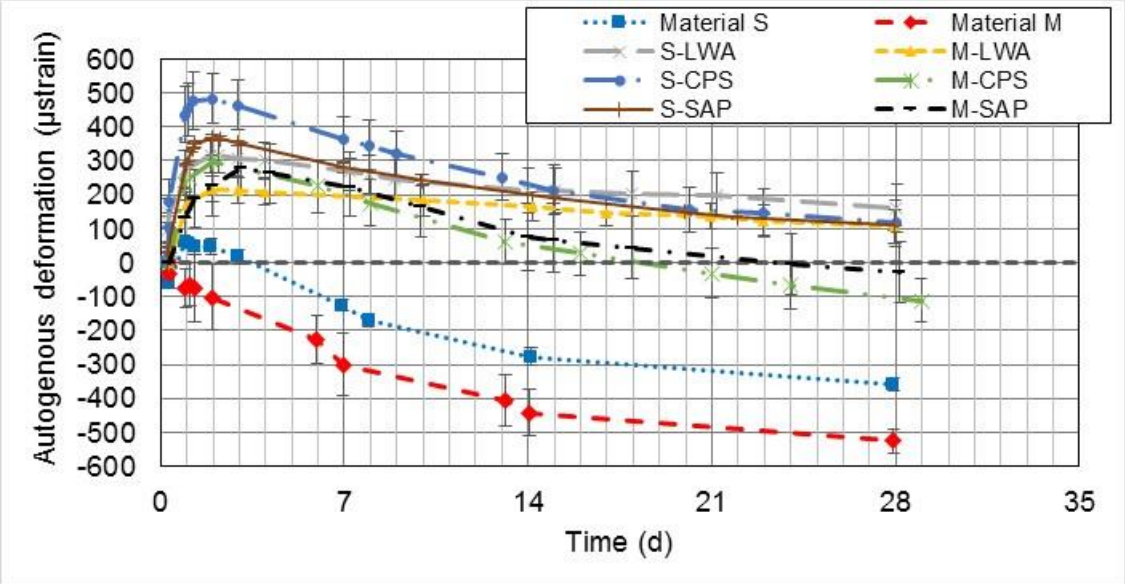

Figure 2. Measured autogenous deformation for the 8 mortar mixtures with and without internal curing. Error bars indicate ± one standard deviation for three replicate specimens.

The XRD results in Figures 3 and 4 differ significantly from those commonly encountered for an ordinary portland cement in the amount of ettringite that is formed at early ages. Likely due to expansive agents added to the mortar formulations, there is a considerable quantity of ettringite that has been generated even prior to the first XRD scan. Ettringite continues to form throughout the first 24 h, as first gypsum and then anhydrite are sequentially depleted as sulfate sources (Figures 3 and 4). The tricalcium silicate (alite) only begins to react in earnest after about 5 h, as supported by the concurrent generation of calcium hydroxide (portlandite), a product of alite hydration. Therefore, for these repair materials, initial setting, occurring between about 2 h and 5 h, is actually caused by ettringite formation and not by the

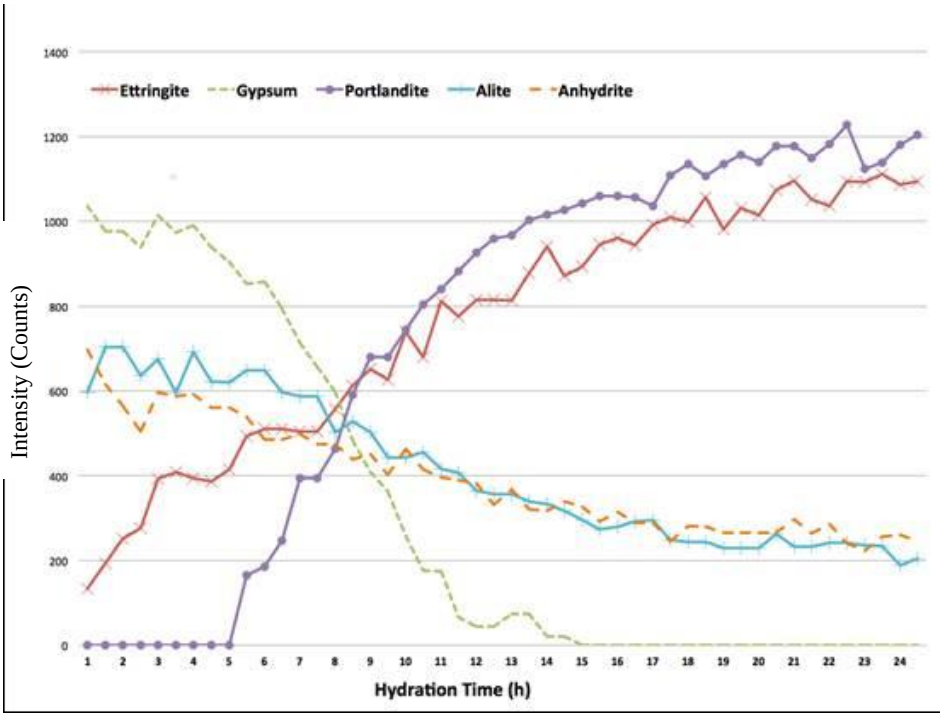

Figure 3. In situ X-ray diffraction measurement of phases present in repair material S (with SAP) as a function of hydration time. A measure of the uncertainty is given by the variability in the counts for alite prior to the precipitation of portlandite at 5 h.

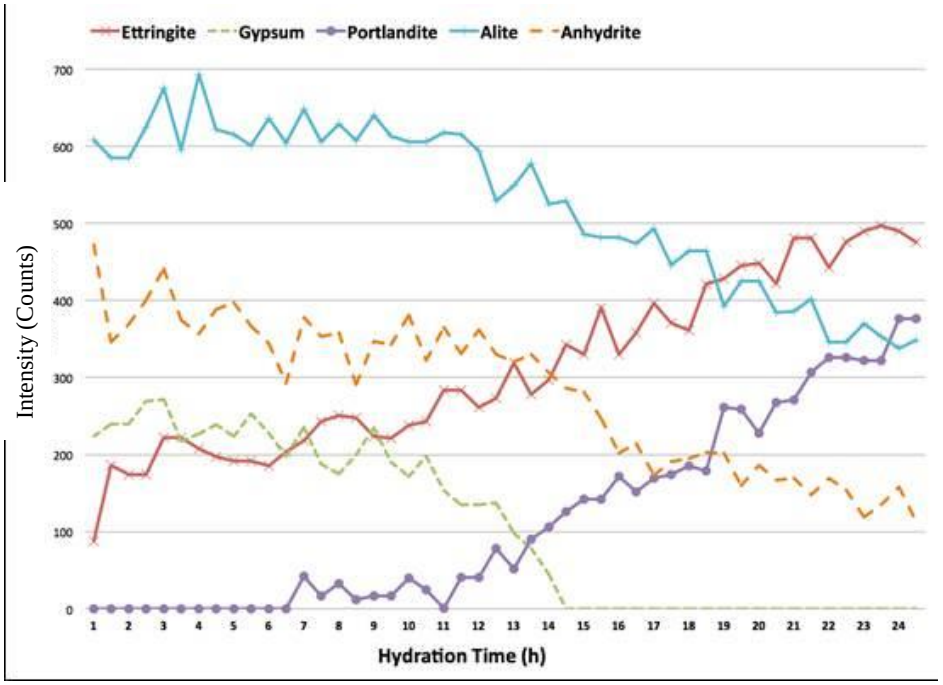

Figure 4. In situ X-ray diffraction measurement of phases present in repair material M (with SAP) as a function of hydration time. A measure of the uncertainty is given by the variability in the counts for alite prior to the precipitation of portlandite at 6.5 h.

production of calcium silicate hydrate gel (C-S-H) as in conventional ordinary portland cement-based materials. At later ages upon total sulfate depletion, this ettringite would be expected to convert to a monosulfate (Afm) phase and it is conjectured that the dissolution of ettringite needles would contribute to the measured autogenous shrinkage in much the same way that their formation contributed to the measured expansion during the first few days. However, additional XRD studies out to later ages are needed to support/refute this hypothesis.

Cusson[12] has suggested that it is not only the measured long term autogenous deformation that is a critical performance measure for early-age cracking, but also the deformation path that has been taken. Thus, he advocates characterizing the autogenous shrinkage by the difference between the maximum measured expansion at early ages (or 0 if no expansion is observed) and the value subsequently obtained at an age of 7 d. The efficiency of an IC agent can then be assessed as the relative reduction in this quantity with respect to that measured on a corresponding system without IC. In the present study, this analysis of Cusson was conducted for ages of 7 d and 28 d and the obtained results are presented in Table 2. For these two repair materials, IC provided by LWA is more efficient than that provided by PCS or SAP, particularly at the age of 28 d. While the less stiff PCS and SAP particles allow significantly more expansion during the first day, they subsequently also allow more shrinkage when this expansion ceases and some of the restraining particles, such as ettringite needles, dissolve from the microstructure. Conversely, the mortars with LWA exhibit a higher modulus (Table 1) to resist deformation and the rigid and more voluminous LWA particles will also provide additional restraint, while the shrinking mortar phase is reduced to occupying only 82 % of the overall system volume in the mortars formulated with LWA for IC (Figure 1), versus about 95 % in those with SAP or PCS for IC.

Table 2: Post-peak to 7 d or 28 d autogenous shrinkage of repair mortar mixtures with and without internal curing as per Cusson[12]

Mixture	Peak-7 d shrinkage (microstrain)	IC efficiency - Reduction vs. control without IC (7 d)	Peak-28 d shrinkage (microstrain)	IC efficiency - Reduction vs. control without IC (28 d)
Material S	186		420	
Material M	301		526	
S - LWA	48	74 %	155	63 %
M - LWA	20	94 %	109	79 %
S - PCS	121	35 %	368	12 %
M - PCS	98	68 %	411	22 %
S - SAP	87	53 %	259	38 %
M - SAP	54	82 %	306	42 %

4.0 CONCLUSIONS

Internal curing can provide a significant reduction in the autogenous shrinkage of repair mortars, as exemplified by the two materials examined in the present study. IC can be provided by different means such as LWA, PCS, and SAP, but each IC agent will have a different influence on the mechanical properties and autogenous deformation of the mortar. For the two repair materials investigated herein, LWA was found to provide the greatest IC efficiency while also having the lowest (detrimental) impact on mechanical properties. Conversely, SAP-based materials induce a retardation of the hydration reactions that along with the increased (empty) porosity/voids remaining after their IC water is released leads to significant reductions in elastic modulus and compressive strength, particularly at early ages.

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